This Page Is Inserted by IFW Operations and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do not report the images to the Image Problems Mailbox.

(19) JAPANESE PATENT OFFICE (JP)

(12) Official Gazette for Laid-Open Patent Applications (A)

(11) Japanese Laid-Open Patent Application (Kokai) No. 58-225003

Internal Office Nos. Ident. Symbols (51) Int.Cl.3

7306-4C 7/00 A 61 K 7915-4B // A 23 J 1/14 7115-4B 109 1/20 A 23 L

(43) Laying-Open Date: 27 December 1983

Request for Examination: Requested

(Total of 8 pages) Number of Inventions: 3

(54) Title of the Invention: A Cosmetic Material Containing

Fermented Soybean Extract

(21) Application No.: 57-107775

(22) Application Date: 23 June 1982

Yoshihiro Chikamatsu (72) Inventor:

11 Shinko-cho

Gifu-shi

Yutaka Ando (72) Inventor:

998 Mitsuzuka-cho

Ogaki-shi

Ichimaru Farkos [phonetic] Company, Ltd. (71) Applicant:

337 Takatomi, Takatomi-cho

Yamagata-gun, Gifu-ken

Specification

Title of the Invention

A Cosmetic Material Containing Fermented Soybean Extract

Claims 2.

(1) A cosmetic material characterized in that it is obtained by the following processes: Fermented soybeans are, in advance, sterilized by heating or sterilized with ethylene oxide gas, after which water is added and they are pulverized, the filtrate then being collected, a solvent that is immiscible with water is added and the material is stirred, after which it is allowed to stand in a cold, dark place, being separated and collected in two parts, the aqueous layer part (A), which has separated, and the solvent layer part (B).

During separation and collection, a solvent that is miscible with water is added to the aqueous layer part (A) and the mixture is stirred, after which it is allowed to stand in a cold, dark place, separating into a precipitate layer part (C) and supernatant layer part (D). These two layer parts are collected separately. The precipitate layer part (C) is an extract that is of high viscosity and of which protein is the principal component and a gummy paste-like elastic solid substance (extract originating from layer C) is obtained by removing the solvent and water from it under reduced pressure. The supernatant layer part (D), from which the solvent is completely removed under reduced pressure, is added to an aqueous solution comprised of one of NaCl, KCl or NaSO4 (sic) and the component that is precipitated by this means is collected. It is next dissolved in strongly alkaline 50% methanol, after which it is filtered and the filtrate is collected and an extract (extract originating from layer D) comprised of a pigment of which the principal component is isoflavone is obtained by concentration under reduced pressure.

Further, the solvent layer part (B), which has been collected separately in advance and which is immiscible with water, is evaporated under reduced pressure, and, after the solvent has been completely removed, is saponified with an alkali, the unsaponified matter is [illegible] by adding ether or n-hexane and then an extract (extract originating from layer B) of which the principal component, a phytosterol, is obtained, the cosmetic material containing, from these respective fermented soybean extracts, the extract originating from layer C alone or both the extract originating from layer B and the extract originating from layer C.

(2) A cosmetic material characterized in that it contains a dry powder that is obtained in the following way. Fermented soybeans are, in advance, sterilized with ethylene oxide gas, after which water is added and the mixture is gently stirred, the string-like viscous substance that is on the surface of the fermented soybeans is peeled off and transferred to the water that was added earlier, the material then being filtered.

Next, an equal quantity of ethanol is added to the filtrate, and, as the mixture is being thoroughly agitated, the aqueous layer that has separated is collected, acetone is used for the extract containing as its principal component protein that was obtained by removing the water by distillation under reduced pressure and the mixture is thoroughly stirred, after which the acetone is removed under reduced pressure.

(3) A powdered cosmetic material characterized in that it contains a dry powder that is obtained in the following way. Fermented soybeans are, in advance, sterilized with ethylene oxide gas, after which water is added and the mixture is gently stirred, the string-like viscous substance that is on the surface of the fermented soybeans is peeled off and transferred to the water that was added, the material then being filtered.

Next, an equal quantity of ethanol is added to the filtrate, and, as the mixture is being thoroughly agitated, the aqueous layer that has separated is collected, acetone is used for the extract containing as its principal component protein that was obtained by removing the water by distillation under reduced pressure and the mixture is thoroughly stirred, after which the acetone is removed under reduced pressure, the cosmetic material as described in Claim 2 of the Claims being dried at the time of use.

3. Detailed Description of the Invention

This invention relates to growing fermented soybean microorganisms (a species of Bacillus subtilis, Aspergillus oryzae) in evaporated soybeans, using the food "fermented soybean" which is obtained by maturation as the starting raw material, obtaining the extract from it and using it in cosmetic materials and food products.

Fermented soybeans are of high nutritional value and have long been provided as a food in Japan. However, they have the drawback that not everyone enjoys eating them because they generate an unpleasant odor (stench) on long-term storage. Moreover, many people keep away from fermented soybeans because of the string-like viscous matter that is characteristic of them.

For this reason, the inventors first conducted various studies with the intention of eliminating the unpleasant odor from fermented soybeans and making an extract of them from which the nutritional components were not lost so that they could be used as food products. As a result, the extract from the soybeans and the principal component was a string-like viscous substance and found to consist primarily of protein. This was not only excellent from a nutritional standpoint but also had an excellent moisture retaining and lubricating effect. In particular, when it was applied to the skin, it exhibited a smooth lubricating action, for which reason it was not only found to have nutritional value but also to be advantageous when used as a cosmetic material. Accordingly, the inventors conducted further studies on its application to cosmetic materials, research was continued on the relationship of extraction methods to yields and humectant effects and this invention was perfected as described below.

We have not found any other previous instances of examples of using extracts of fermented soybeans in cosmetic materials or examples of using such extracts as food products. Because the extraction method from fermented soybeans in this invention involves a simple procedure, it can be anticipated that new fields for its utilization will be opened up. Even people who have not eaten fermented soybeans could easily anticipate applications other than cosmetic products for example beverages prepared from their extracts, as mixtures with suitable vehicles and other nutritional agents by processing them as granules, tablets or fillings for gelatin capsules.

[Example 1]

Fermented soybeans were, in advance, sterilized by heating, water was added and they were pulverized to form a gruellike substance. Next, the gruel like substance was filtered and the filtrate was collected. This filtrate was a viscous liquid. Any one solvent selected, for example, from chloroform, esters such as isoamyl acetate, isopropyl acetate and isobutyl acetate, alcohols such as n-hexyl alcohol and decyl alcohol and hydrocarbons such as n-hexane, n-heptane, benzene, petroleum ether and cyclohexane, which are known solvents immiscible with water, was added in a proportion on the order of 10 to 50% to the filtrate and the mixture was allowed to stand for about a full day in a cold, dark place as it was being stirred. By this means, it was separated into an aqueous layer part (A) and a solvent layer part (B). The A (layer) was collected and separated, after which one solvent miscible with water selected, for example, from ethanol, methanol, acetone and propyl alcohol was added to layer (A) in an amount on the order of 30 to 90% relative to layer A and the mixture was stirred. After stirring, the mixture was allowed to stand for about a full day in a cold, dark place, with the result that it separated into a precipitate layer (layer C) and a supernatant layer (layer Layer C (the precipitate) was separated and collected. This precipitate, an extract, contained as the principal component a protein of high viscosity. This extract was then gradually transformed to a gummy paste-like elastic solid by removing the solvent and water under decreased pressure. When it was used in cosmetic materials and foods, it was used as a suspension (emulsion) dispersed in water.

[Example 2]

Layer (B), which had been separated in the process of Example 1 as described above, was distilled under reduced pressure, with the solvent being completely removed. When this was done, fats and oils remained and the characteristic stench (foul odor) of fermented soybeans was present. They

were saponified with an alkali, after which the unsaponified matter was removed by adding ether or n-hexane and an extract of which the principal component, a phytosterol was obtained. The foul odor was thus eliminated from the substance obtained in this process.

[Experiment 3]

Layer D, which had been separated in the process of Example 1 as described above, was distilled under reduced pressure, with the solvent being completely removed, and it was added to an aqueous solution prepared with any one of NaCl, KCl or Na₂SO₄, the component that was precipitated by this means was separated and collected and then dissolved in weakly alkaline 50% methanol, after which it was filtered, the filtrate was collected and an extract of which the principal component was isoflavone and which was comprised of a pigment component was obtained.

The total yields of the extracts that were obtained in the aforementioned Examples 1 to 3 were on the order of approximately 250 to 300 g from 5 kg of fermented soybeans. Of these, the extract of which protein was the principal component (the extract obtained in Example 1) accounted for most of the yield, or 86 to 90%. The remainder was comprised of the extract of which a phytosterol was the principal component (the extract obtained in Example 2), which accounted for 3 to 6%, and the extract comprised of pigment components of which isoflavone was the principal component, accounted for 1 to 3%.

The extracts obtained in the aforementioned examples can be used independently in cosmetic materials and foods. In particular, the extract of which the principal component is protein that was obtained in Example 1 exhibits a high viscosity of about 50 to 80 cps in a concentrated liquid At this viscosity, moisture retention and lubricity Further, in mixed solutions of ethanol and are exhibited. water, it is miscible with the extracts obtained in Examples 2 and 3, with lubricity being increased. Specifically, it does not have a smooth sticky feel and has a humectant Therefore, it can easily be used in cosmetic effect. materials and foods. It also has a good taste. In order to bring about the characteristic body of fermented soybeans, it is preferable to make combined use of the extract of which the principal component is a sterol and of the extract comprised of pigment components of which isoflavone is the principal component, which were obtained in Examples 2 and 3, rather than only the extract of which protein is the principal component.

Basically, when suitable fragrances and refrigerants are added and the materials are diluted with water or ethanol, they can be used as simple toilet water and beverages. When the viscosity number at this time is regulated to the order of 10 to 30 cps on the basis of the quantity of extract added or its content, there is a good slippery feeling

characteristic of fermented soybean extracts. The humectant and slipperiness effect is similar to that of [illegible] gum polysaccharides such as hyaluronic acid. It is characteristic that there is no sticky feeling. In short, the humectant effect differs from that attributable to proteins originating from animals such as collagen and albumin in that a smooth, non-sticky touch is obtained.

Next, the fermented soybeans that were the raw materials in the aforementioned examples were heated and sterilized in advance, after which the extraction method was used. Decreases [antecedant not given-Translator] were found as the time required for heat and sterilization in order to obtain high viscosity extracts was prolonged. For this reason, in order to obtain high viscosity extracts, an additional study was made of the distillation procedure. short, various studies were conducted of the sterilization process and of the intermediate processes and final process during extraction. In all cases, it was found that workability was difficult due to proliferation of the bacteria and that sterilization in advance of the fermented soybeans, which are the raw materials, was the best method Moreover, when sterilization with ethylene for extraction. oxide gas was studied as a method with which there would be no effect on viscosity in the sterilization procedure and which would be advantageous on an industrial production scale, it was found that, over the course of time, extracts of higher viscosity could be obtained and that yields could be increased.

[Example 4]

Amounts of 100 g each of fermented soybeans were introduced into a pack for Hi-zex film sterilization of 28 cm in width and 15 cm in width, ethylene oxide gas was sealed into it and it was allowed to stand for specified times as shown in Table 1, after which bacteriological tests (agar plate dilution method) were performed. After sterilization effectiveness was evaluated, amounts of 1000 ml of purified water were added to fermented soybeans that had been subjected to the bactericidal effects of the ethylene oxide They were then stirred at a temperature of 15 ± 1°C and were further stirred slowly for 1 hour at a rotation rate of 100 rpm, with a viscous substance material similar to a string-like substance on the surface of the fermented soybeans being the principal material extracted. viscous liquid that was cotained was filtered (Toyo Filter Paper No. 65) by suction filtration. Next, the same volume of ethyl ether was added to the filtrate and the mixture was thoroughly agitated, after which the aqueous layer that was separated was collected. The water in the aqueous layer was removed by distillation under reduced pressure and a solid substance (extract of which protein was the principal component) was obtained. Next, the solid substance was thoroughly washed with acetone, the acetone was removed under reduced pressure and a dry powder was obtained. yields were as shown in Table 1. It was found that yields

increased and viscosity also increased over the course of time in the ethylene oxide sterilization treatment. Solubility in water also increased by comparison to substances extracted from fermented soybeans that had been subjected to heat treatment.

Table 1 shows the yields and viscosities of extracts of which the principal components were proteins that were extracted using fermented soybeans as a result of ethylene The solubility rate (%) was determined oxide sterilization. for a transparent solution obtained by weighing out amounts of 1 g of extract (extract obtained by Example 4) of which protein was the principal component, introducing the extract into 250 ml of purified water at 20°C and stirring the mixture for 1 hour at a rotation rate of 300 rpm, with an emulsified protein being formed, after which this liquid was subjected to forced filtration using an 0.8 micron membrane In short, the extracts of which proteins were the principal components that were extracted from the fermented soybeans and that were obtained in Example 1 or Example 4 were thoroughly dispersed in water and appeared as a white There are extremely few liquids or milky brown dispersion. that appear as transparent aqueous solutions, i.e., in which the protein components are completely soluble in water. solubility rates shown in Table 1 are for these watersoluble proteins. The same is true for viscosity. short, it was found that the protein component when it is emulsified and dispersed in water constituted the principal component with respect to the characteristic viscosity of fermented soybeans. The upper limit of the highest value of the viscosity exhibited by the dry powder itself of the extract of which protein was the principal component as obtained in Example 4 when it was dispersed in water was on the order of 100 cps. On the average, it showed a peak at 80 to 90 cps. As a result, use of fermented soy beans that have been subjected to sterilization treatment with ethylene oxide gas is not only more bactericidally effective against Bacillus subtilis than fermented soybeans subjected to heat treatment, but, at the same time, the amount of viscous material that was extracted from the fermented soybeans also increased as treatment time was prolonged. At the same time, it was ascertained that dispersibility and solubility The cause of this is believed to be that were increased. the ethylene oxide becomes attached to the Bacillus subtilis, displaying a bactericidal effect and that it also becomes attached to the fermented soybean protein, for which reasons solubility is increased and there are also increases in yields.

(Table	1)	Yields	of	Proteins When	Fermented	Soybeans	Sterilized
•		with E.	Ο.	Were Used			

(h)	Bacillus subtilis, ordinary bacteria (number)	Mold, yeast (number)	Yield (%)	Solubility (%)	Viscosity (cps)
Untreated	10 ⁷ /g	0	2.4	0.04	2.4
12	10 ⁷ /g	0	3.1	0.08	4.7
24	3 X 10 ³ /g	0	3.1	0.09	4.5
48	2 X 10 ⁴ /g	0	3.3	0.10	4.9
72	5.6 X 10 ³ /g	0	3.2	0.09	4.7
96	3200/g	0	3.4	0.09	4.6
120	550/g	0	3.3	0.11	5.0
144	108/g	0	3.4	0.10	4.6
168	30/g	0			
240	20/g	0			

We shall now describe the uses of extracts obtained in Examples 1 to $4\,\cdot$

The extracts of which proteins were the principal components that were obtained in Example 1 and 4 can be used independently and do exhibit good taste so that they can be used as foods. They can be added to various processed food products as humectant agents and extracts, of which the principal components are proteins, may be dispersed in water to adjust the viscosities to the order of 1 to 20 cps so that they can be used in cosmetic products and beverages. When the extracts, of which phytosterols are the principal components as obtained in Examples 2 and 3 are added to and thoroughly mixed with the extract containing a pigment component of which isoflavone was the principal component and with the extract of which proteins are the principal components as obtained in Examples 1 and 4 , the humectant action is different from that of extracts in which proteins are the principal components. In terms of taste, the characteristic body of the fermented soybeans was increased and the lubricating effect was increased.

We shall bow present examples of formulations.

(Reference Examples of Formulations)

(1) Beverage

Solution obtained by dispersing the extract, of which proteins were the principal component, obtained in Example 1 or 4, in water and a small quantity of ethanol and adjusting the viscosity to 50 cps

-	
	1 to 30%
•••	0.2
Lactic acid	0.2
	0.9
Citric acid	
Sweetening agent	3 - 10
	0.1
Preservative (paraben agents)	0.1
	Suitable quantity
Fragrance	-
Purified water to make a total quantity	of 100.

(2) Cosmetic Material (Lotion)

Solution obtained by adding water to and dispersing the extract of which the principal component was protein obtained in Example 1 or 4 and the viscosity of which was adjusted to 30 cps

	5.0%
 Whale tallow	2.0
Beeswax	16.0
Liquid paraffin	46.5
Cetyl alcohol	2.0
Purified water	26.8
Borax	1.0
Fragrance and paraben (methyl)	Suitable quantity

(3) < Vanishing cream >

Stearic acid	16.0%
	2.0
Sorbitan monostearate	1.5
Polyoxyethylene sorbitan monostearate	1.0
Extract of which protein was the	

Extract of which protein was the principal component that was

obtained in Example 1 or 4 3.5-4.5
Propylene glycol 10.0
Fragrance and paraben (methyl) 0.2

Purified water to male a total quantity of 100.

(4) < Toilet Water >

Ethanol	9.0%
Lactic acid	0.2
Citric acid	0.9
	4.0
Sorbitol	0 11-7

Fragrance, colorant, preservative Suitable quantities

Aqueous solution obtained by mixing 0.8% of the extract containing pigment component of which isoflavone was the principal component obtained in Example 3 with 20% of the extract of which protein was the principal component obtained in Example 1 or 4

Purified water to make a total quantity of 100.

(5) < Cold cream >	
Beeswax	10.0%
Gelatin	10.0
Vaseline	15.0
Lanolin	5.0
Liquid paraffin	17.5
Olive oil and rice germ oil	10.0
offive off and fice germ off	
Extract of which protein is the	
principal component obtained in	3.0
Example 1 or 4	5.0
Extract of which the principal	
component is a phytosterol	
obtained in Example 2	0.5
Extract of which the principal	
component is isoflavone obtained	
in Example 3	0.2
Oryzanol	1.0
Purified water	22.7
	1.0
Fragrance	0.3
Preservative	

[Humectant action]

Next, we shall consider the humectant action of the protein obtained in Example 1 or 4. It was dispersed in advance in water and a solution was obtained, the viscosity of which was adjusted to the order of 30 cps. This solution was further diluted 20 times to make the test solution. The quantity of water that escaped from the solution was found by the gravimetric method to the point that a constant volume was reached at a relative temperature that had been set using a constant temperature and constant humidity tank. The samples were compared using an aqueous solution containing 5% of sodium pyrrolidone carboxylate. The results, as shown in Figure 1, indicate that they had the same humectant action. On the other hand, the toilet water, as indicated below was made using the solution diluted 20 times that was used in the aforementioned test. This toilet water and toilet water to which nothing was added were used in studies of feel on use. Application tests on the skin were carried out

using 40 women as subjects. The results are shown in Table 2. As can be seen, there was no sticky feel, there was a good smooth touch, there was a superior lubricating effect and a clean feel on use.

(Formulation: Toilet water)	
Ethanol	9.0%
Lactic acid	0.2
Citric acid	0.9
Sorbitol	4.0
Dilute solution of extract	
(viscosity, 3 to 5 cps)	8.0
	0.1
Fragrance	0.1

Purified water to make a total quantity of 100)

(Table 2) Use Response Tests of Toilet Water Containing Fermented Soybean Extract

JOyk	Can Datace				T
	Content	Poor	Ordinary	Fairly good	Good
Transparency	Not added	0	2	32	6
[poor legibil- ity - Trans.]	Added	0	20	19	1
Cleanness of	Not added	0	6	31	3
skin	Added	0	5	14	21
Smooth feel of	Not added	17	20	3	0
skin	Added	0	8	12	20

[Safety]

It was presumed that there are no problems of safety associated with extracts based on this invention as the starting raw material is fermented soybean which is supplied as a food. However, for the sake of precaution, the extracts of which protein was the principal component, obtained in Examples 1 and 4, were studied by cral administration in mice. The extracts were dispersed in purified water and solutions were used that were prepared of viscosities of approximately 30 cps. The LD_{50} values were less than 40 ml and it was concluded that there were no problems and that the materials were of high safety. In addition, primary irritation tests were performed using the aforementioned solution. Forty-eight hour patch tests were performed with the same 40 women who participated in the use response test described above. No abnormalities such as erythema were found.

A further point to which attention should be drawn is that the extracts of which protein was the principal component that was obtained in Example 1 and Example 4 have both a humectant lubricating actin and a tyrosinase activity inhibitory action.

When further interest was drawn to this point and we conducted follow-up tests of the extracts obtained in Examples 1 through 4, these actions were found for all of the extracts that were obtained except for that of Example 2.

Consequently, fermented soybeans are advantageous as substances with which both a humectant lubricating action and a beautifying-whitening action on the skin can be expected. Table 3 shows the results of in vitro studies of the melanin pigment production inhibiting action exhibited by the fermented soybean extracts obtained in Examples 1 though 4 of this invention.

The reaction system in the experiments was comprised of 0.5 ml of L-tyrosine (1.0 mg/ml), 2.0 ml of phosphate buffer solution (pH 6.8), 2.0 ml of distilled water or inhibiting agent solution (extract), 0.05 ml of Cu** ions (1% solution) and 1.0 ml of tyrosinase (1 mg/ml). The reaction was allowed to proceed for 60 minutes in a constant temperature tank at 37.5°C. After it was concluded, absorbance at 640 nm was measured with a spectrophotometer and the production rate was calculated. Ascorbic acid was used as the comparison test substance.

(Table 3) Melanin Production Inhibiting Action of Fermented Soybean Extract

Soybean Excrace		
Test substance (2% inhibiting agent solution, content in solution)		Inhibition rate (%)
Purified water		0
Vitamin C	0.5	97.5
Extract of which protein is the principal component in Example 1	5.0	62.1
Extract of which phytosterol is the principal component in Example 2	1.0	12.6
Extract of which isoflavone is the principal component in Example 3	0.5	68.0
Example of which protein is the principal component in Example 4	5.0	67.4
Mixture of extracts obtained in Examples 1 to 3 at the yield ratios	5.0	63.6

With the dry powder of the extract obtained in Example 4, dissolved at the time of use, a filling of good feel and lubricating characteristics was obtained.

In short, it is a method in which a dry powder is mixed with a cosmetic base material individually or with another powder and in which the mixture was dissolved separately using an aqueous solution, a known toilet water, emulsion or cream.

As shown below, when a powdered cosmetic material was made and toilet water was used, both were collected on the flat of the hand. The powdered cosmetic material was kneaded with the fingertip and was dissolved, being used in that way.

(Powdered cosmetic material)

- (1) Dry powder obtained in Example 4 1 70 % Vitamin C 0.3 - 50 Purified water to make a total quantity of 100.
- (2) Dry powder obtained in Example 4 90 95% CMC or alginic acid 5 10
- (3) Extract obtained in Example 2 1 2 %
 Extract obtained in Example 3 0.3 1
 Extract obtained in Example 4 80 95
 Oryzanol (fine powdered product) 0.2 0.3
 Vitamin C 1 3
- (4) Dry powder obtained in Example 4 95 %
 Aloe polysaccharide powder
 (Peragel 200) 0.3
 Vitamin C 3 4
 Oryzanol (finely powdered product) 0.6 2.7

The aforementioned powdered types of cosmetic materials are all of a high degree of hygroscopicity (moisture absorbing capacity), for which reason they should be packaged in hermetically sealed containers for solutions. They may be kept in single batches or divided into packets of 0.1 to 2 g. They can also be compounded in foundations and packs. In this case, they may also be used in combination with silk packs and with low molecular weight peptides of silk. In making cosmetic materials, the extracts obtained in Examples 1 through 4 can be mixed in advance in combinations as desired with formulations of other cosmetic base materials and solutions may be made by dispersing and dissolving them in solvents such as water, water and ethanol or water, ethanol and polyols. This is convenient for compounding them.

On the other hand, in the extraction processes in Examples 1 through 4, the fermented soybeans are used after they have been sterilized in advance by heating or with ethylene oxide gas and the substances that are subjected to the extraction treatment operation can also be extracted using fermented soybeans that have not been sterilized in advance. At this time, in the extraction treatment process, the extraction treatment is performed as far as possible at about 20° or at a lower temperature than that. In particular, extracts of which the principal component is protein are concentrated under reduced pressure to make a powder or are placed in a freeze-drier to make a powder, after which, in the final process, the powder is

subjected to sterilization treatment with ethylene oxide gas, by which means it is rendered sterile.

4. Brief Explanation of the Figure

Figure 1 is a graph showing the humectant action of extracts obtained in Example 1 or Example 4 of which the principal component is protein that were made into aqueous solutions and the viscosities of which were adjusted to the vicinity of approximately 3 to 5 cps.

- 1: Dilute solution of extract of this application
- 2: Aqueous solution containing 5% of sodium pyrrolidone carboxylate.

Applicant: Ichimaru Farkos [phonetic] * Company, Ltd.

(Representative) Yutaka Ando [seal affixed]

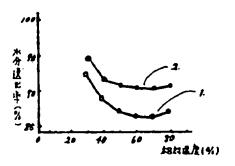


Figure 1

[vertical axis]: Water escape rate (%)

[horizontal axis]: Relative humidity (%)

*Translator's Note: Transliterated phonetically from the Japanese. As such, the spelling may differ from other transliterations.

(P) 日本国特許庁 (JP)

①特許出願公開

母公開特許公報(A)

昭58-225003

昭和58年(1983)12月27日

漢別記号 庁内整理番号 7306─4C

7306--4 C 7915--4 B 発明の数 3 7115--4 B 審査請求 有

(全8頁)

60納豆抽出物含有化粧料

2019

夏 昭57--107775

②出 順 昭57(1982)6月23日

岐阜市新興町11番地

109

① 発明者安護裕

大垣市三城町1996番地

の出 顧 人 一丸フアルコス株式会社

岐阜県山県郡高宮町高宮337番

地

и**д # 8**

上毎明の名称

的互动出物含有化粧料

2.特許請求の報題

(1) 納豆を、あらかじめ加熱装置又はエテレンオャサイドガス減量させてから、水を加えて破砕させたのち、その油板をとり、水に洗和しないところの容易を加えて、提拌したら、冷暖所に影響させて、分離された水層部〔A〕と、治療層級〔B〕の二つに分取する。

かの一種制による水溶散に添加し、これによつて折出した成分を取りだし、次に縄 T ルカリ性 3 ロ 5 ノ ナノールに削解させてから、減温して減敏を取り、減圧過糖により、イソフラボンを主体とする色素成分からなる抽出物(D 層由来始出物)

(4) 前型をあらかじのユチレンオキナイドガス被害してから、木を加えてゆるやかに復粋し、 前型表面にある、糸引状の筋関係を、飼配さ

科教育58-225093(2)

せて、先に加えた水に移行させてから、調査する。

次に、減液に対して、商量のエタノールを加え、十分に摂動したら、分離した水層部を取りだして、減圧高額によつて水分を留まして得られた、蛋白質を主体に含有する抽出物に対し、さらにアセトンを用いて、十分洗浄してから、減圧下でアセトンを留去させて得られた、乾燥粉末を含有することを特徴とする化粧料。

3.発明の評潔な説明

、胎束化脏鼻。

本先明は高度大豆に約豆腐(Bacillus Subcilis の一種、Aepergillus Orysas)を繁殖さ せて、熟成して持られる、食用「約弦」をスタ ート 原料となし、これをもとに、その抽出物を 得て、化粧品質をはじめ、食品質に応用するこ とに属するものである。

にフォトンを用いて、十分洗浄してから、減圧 下でフォトンを留安させて得られた、乾燥粉末

を含有する、特許親求の範囲、第2項記載の化

粧料が、用時避解して用いることを特徴とする

納豆は、栄養質の高い食品の1つとして、わが国においては、古くから食用に供されてきた

(3) 納豆をあらかじめエチレンオキサイドガス蒸掘してから、水を加えてゆるやかに抜粋し、納豆変面にある、糸引状の粘膜物を利能させ、加えた水に谷行させてから、油品する。

次に適能に対して、同量のエタノールを加え 、十分に振動したら、分離した水層部を取りだ して、減圧蒸射によつて、水分を留去して得ら れた、蛋白質を主体に含有する抽出物に、さら

が、しかし欠点としては、長額額の保存は興臭 (タヤミ)が発生するために、すべての人々が 好んで食べるには至つていなかつた。又、納豆 特有の糸引状の粘性物は、これもまた改造する 人々も多く、したがつて納豆自体は、咿好的な 側面をもつた食品として、現在に至つている。

(実施引1)

利豆を、あらかじめ加熱能質し、これに水を 加えて破砕し、カニ状物となしたら、次にカニ 状物を強適して、その適度を得る。この適度は 枯萎な液であるが、次に水に変和しないところ の公知な溶板である、たとえばナロロホルム、

新聞号58-225903 (3)

お銀イソアミル、 酢酸イソプロピル、酢酸イソ プナルなどのエステル頭、コーベキシルアルコ ール、ナシルアルコールなどのアルコール無い ューヘキャン、ューヘブタン、ベンゼン、石油 ユーナル、シナロヘキサンなどの製化水素から 遊びだした、いずれかの一種の落葉を、濃葉に 対して10~505程度を加えて、よく提許さ せてから、一星夜程度、冷暖所に夢聞し、これ によって木海路(A種)と油柴海部(エ層)と に分離し、1月を分取した後に、1月に対し水 に裏和する消滅である、たとえば、公知なエタ ノール、メタノール、 アセトン・プロピルアル コールなどから達んだ一種繋を、A着に対して 30~90%程度を加えて提押する。提押機は 一昼夜昼夜、冷酷所に野難し、沈æ巻層〔c層] と上数数階(D層)に分離し、0層を分取し 、沈蒙物を取り出す。このものは、名性の高い 釜白堂を主体とする旅出物である。 この旅出物 は、さらに越圧下で溶膜や水分を飲ますること により、次第にゴムノリ状の弁力性のある国形

状物となるが、化粧料や食品に関いるときは、 再度、水に分散した繊維 (乳状) 物を削いる。 (実施例2)

上記した実施例1の工程中で分離した3層を用い、これを減圧集留して完全に兼談を散ますると、治監獄が気留する。このものには、納豆特有のチャミ(異臭)が移行しているも、これをアルカリでケン化させてから、その不ケン化物を、エーテル又は3一へキサンを加えて抵取し、フィトステロールを主体とする独出物を得た。この工程を得たものは、異臭が歌会されている。

(実施何5)

前記した実施例1の工程中で分離した3月を用い、これを献圧振信して完全に答案を執金し、 8401、 842804の内、いずれかの一種に 16 水溶液に添加し、これによつて新出された 成分を分取し、次いで到了ルカリ性5 0 5 メリノールに溶解させてから、通過して過敏を取りだし、減圧機能することによつて、イソフラギ

ンを主体とする。色素成分からなる輸出物を得た。

以上の実施例1~3で得られた各独出物の総収量は、結正3年から約800~3009程度であつた。その内、 近白質を主体とする始出物であった。その内、近白質を主体としてもからで、86~908をしめ、 残りはフィトステロールを主体とする独出物(実施例2で再た治出物)が3~68、イソフラがンを主体とする色素成分からなる抽出物は、1~3%程度であった。

自記実施例で得られたエキスは、それぞれ単立で、化社科や食品に用いることも可能であるすが、とくに実施例1で得た、姿命白を主体を要する。 はははは、 込度が高く、 漁輸した 変を生体を示する の 一 8 0 で 9 8 前後の 数 位 を示す。 このを 8 0 で 9 8 前後の 数 位 を示す。 このを 8 0 で 2 で 4 で 4 で 4 で 5 の 5 な 5 を 5 の 5 で 5 の 5 か 5 で 6 を 6 で 7 で 7 で 8 の 7 で 8 の 7 で 8 の 7 で 8 の 7 で 7 で 8 の 7 で 8

有するもので、化粧料や食品にも用いられやすい。又、味も良好であり、純益特有のコクを出すには、蛋白質を主体とする抽品物のみよりも、実施例をやって得たところのステロールを主体とする他出物や、イソフラボンを主体とする。 色素能分からなる抽出物を併用すると良い。

次に、前記の実施例においては、近斜である 前豆を、あらかじめ加熱液面してから抽出する

特別電58-225003(4)

方法を採用したが、高粘度な雑出物を得るには 、加熱減量に要する時間が長くなるほど、低下 することがわかつた。そこで、高粘度な抽出物 を得るために、最終操作について、さらに再換 針を加えることにした。つまり、減難値称につ いて、拍出の際の中間工程や、最終工程などに おいて、祖々の検討を加えてみたが、いずれも 作業性は超世の増盤などにより困難であり、あ らかじめま料となる前豆を設置させ、これをも とに抽出する方法が最善であることがわかつた 。そこで、さらに厳国操作について、粘度に影 響しないで、工業的主産養装においても有利な 方法について、エチレンオキサイドガス装置を 試みたところ、直通時間により、むしろ粘度も 高い抽出物が得られると共に、さらに、収量的 に増加することがわかつた。

(雪集例4)

・納旦各点1009を用い、概要8m×模15 mのハイゼックスァイルム議會用ペックに入れ ・、エチレンオキサイドガスを第1妻に示すごと

肉を示した。

第1表は、ユチレンオキサイド被目による。 前豆を用いて拍出された。蛋白質を主体とする 抽出物の収量及び粘度についてみたものである 。尚、治解率(5)は、西白質を主体とする抽 出售(実施資金で得られた抽出物)各々19を 精界し、200の需要水250半中に入れて複 作し、回転数300 rpm で1時間行い、乳周属 白浪となした後、この食体を 0.8 も タロンのメ ンプランフィルターにより、強調議論させて、 得られた透明な辞根について調定したものであ る。つまり、実施例1又は実施例4にだいて得 られるところの、め豆から独出された蛋白質を 主体とする始心物は、木によく分数するもく乳 白色又は乳費色の腰房食を呈するものであるが 、透明な水溶散 呈した放体、すなわち完全に 水に可差性の蛋白質部分は、ごくわずかであり 、第1世に示す治療率は、この水溶性蛋白質に ついて示したものであり、粘度についても、何 はによるものである。つまり、前豆の有する特

く、所定時間対入して放散したのち、組儡試験 (東天平板世駅法)を行い、装御曲景を報定し たのち、それぞれのエテレンオキサイドガス袋 直済的互について、 荷賀水1000㎡を加えて 、当度16±1でで最終させ、100 rpm の征 転載で1時間、雄やかに推荐させ、前立表情に ある糸引状の蛋白質を主体とする、粘層物を始 出した。待られた私機な故を、後引進基故によ り、雑紙(京洋雑紙高68)で雑画する。次に 、通復に対して、同量のエチルエーテル 十分に製量してから、分離する水量を掘り出 して、これを禁圧蒸留によつて、水を留金させ て何形物(蛋白を主体とする益出物)を得る。 次に、アセトンで回影物を十分洗浄し、減圧下 でアセトンを留法させて、 花集粉末を得る。収 量は、それぞれ毎1後に示すごとくであつた。 エナレンオーサイド設置処理における時間の経 過と共に、収量が増加し、粘度も上昇すること がわかつた。又、水に対する前無性も、加熱筋 通した納見から抽出したものに比べて黒きる45

有の粘度は、水に乳毒分数するところの蛋白質 年分が、その主体をなしていることがわかつた 一方、矢施背もによつて得られたところの、 蛋白質を主体とする並出物の乾燥粉末自体が示 す粘度は、これを水に分散させて、一番高い長 他の上段は、100 cps 程度となり、半角的に は 8 0~ 9 0 cpe にピーナを示す。この結果、 紅熊処理した前豆を用いるよりも、エチレンォ キサイドガス被害処理した納豆を用いる方が、 枯草目に対する収置に有効なばかりでなく、例 時に処理幹間の発長は、これにともなつで前並 から拍戯される、粘稠な蛋白質の重も増加する ことがわかつた。阿弉に水に対する分散性及び 海岬性も肉上することが 幇明した。この裏因は - ステレンオキサイドが枯草層に開加して、投 舞効果を表わすと共に、 納豆蛋白にも関加して 、 これがために治療性が向上し、収量的にも増 口を示したものと考えられる。

(部1表) 8・0・倉田による納里を用いた、蛋白質の理能など

7	総単数・一位 単数 (数)	∌ ₹ - # 8 (3 2)	(多)	(E)	tope)
*#2	1070	0	,14	0.04	2.1
12	1070	0	2.1	0.00	4.7
24	3×105	0	1.1	0.09	4.6
44	2×10/9	0	7.2	0.10	4.0
71	3.0×103/1	0	1.8	0.07	4.7
••	3200/9	. 0	7.4	0,01	4.4
120	130/1	. 0	7.3	0.11	B. 0
144	L00/1	0	3.4	0,10	4.6"
3.00	30/1	G			
140	20/9	G		<u> </u>	

次に実施例1~4で得られた各々の抽出動に ついて、その用途費に関して述べる。

実施例1及び4で得られた蛋白質を主体とす る物は物は、そのまま単数でも味も皮好であり 、食用とすることもできる。又、保理剤として も各種の加工食品中に参加して用いることも出 患るが、化粧料や飲料では、蛋白質を主体とす

将貨水をもつて全量を100とする。

(a) 化粧料 くローション〉

実験例1又は4で券た長白質を主体とする船 消物を、水を加えて分散させ、粘度を S O sps 5.0 ≸ 2.0 1 6.0 セチルアルコール ・・・・・・・・・・・・ 1.0 香料及びベラベン(メチル)・・・・・ (3) 〈ドニシングクリーム〉 ステアラン酸・・・・・・・・・・・・・・・ 1 6.0 \$ ソルビタン・モノステアレート・・・ ポリオキシエチレンソルビラン モノステアレート・・・・・・・・ 実施別1又は6で、た、蛋白質を主体とする類。

特別電58-225803 (6)

る始出物を、水などに分散させて、粘度につい て1~20 cye 程度になるように興奮して耐い ると良い。さらに、実施料1~3で得られたと ころのフィトステロールを主体とする前出物や 、イソフラギンを主体とする色製成分を含む薬 出物とは、実施費1や4で得られたところの値 白質を主体とする抽出物とは、よく運和す の で一葉に単独して用いると、保護作用は仮白質 を主体とする抽出物と変らないが、味は的豆幹 方カコナが増し、滑性効果は向上する。 以下に 、処方例を示す。

(参考基方例)

-(1)飲料

実施例1又は6で券と仮白賞を主体とする抽 出袖を、木と少量のエメノール中で分散させ、 粘度を50 ape に異常した治療・・・ 1 ~ 30g # 防腐剤(バラベン制)・・・・・・・・・

書料及びパラベン(メチル)・・・・・ 精製水をもつて全量を100とする。

(4) 〈化粧水〉 9.0 S x 4 / - A Ó. Z 香料、詹色料、防腐剂 ****** 通 実施男1又は4で得た仮白質を主体とする抽 出物と0%に、実施例3で得たイソフラギンを 主体とする色質点分を含有する軸貨物の65分で、

通和した水溶液・・・・・・・・・・・・ 4 0.0 特観水をもつて、全量を100とする。

(3) 〈コールドタリーム〉		
₹ 7 B 7 ······	1	0.0 \$
* V 9 > · · · · · · · · · · · · · · · · · ·	3	0.0
7 + 9 >	1	3.0
3 / 9 >		3.0
進動パラフイン ************************************	1	7, 5

待興町58-225983()

水溶液を用いて比較したが、その底管筋条は霧 1 四に示すことく、ほぼ何じ保証作用を有して いることがわかつた。一方、前記試験に用いた 20増長収載を用いて、次に示す化粧水を作り 無路的の化粧水と、使用器について、40名 の女性を針章にして、肌に対する他和試験を行 つたが、その結果は第2世に示すことく、べた つき感がなく、ナベナベした感触が臭好で潜性 効果に優れ、さつばりとした世別感が持られた

(格方:化粧水)

1.0

I	*	1	-	*	• •	• • •	••	•	• •	•	••	••	• •	• •	•	. 0	1
я,			2	•	• • •	• • •	••	•	• •	••	• •	••	••	• •	•	0.2	
. 2	3 .	ン		•	• • •	• • •	• •	• •	• •	••	••		••	••	•	g. 9	
y	r	۲	7	۲	••	• • •	••	•	• •	• •		••	••	••	•	4.0	
抽	出	*	Ø	#	ĸ	板	(粘		3	~	6	•	P) ••	8.0	
•			Ħ	•	• •	• • •	• •	•	• • •	••	••	• •	• •		•	6.1	
	***		*	T	全		1	0	0	٤	†	ē	•				

オ リー ブ 袖 又 は 米 胚 芽 油・・・・・・・ 1 0.0 実施例1又は4で持られた蛋白質を主体とす 実施例えて待られたフィトスナロールを主体 実施例まで持られたイソフラボンを主体とす オリザノール ・・・・・・・・・・・・ 1.0

2 2.7 **#** ••••••

0.3

(保護作用)

次に、実施例1又は6で持られた蛋白質の保 展作用についてみると、あらかじめ水で分散さ せて、結成を S O ops 固度に興難させた治療を 財い、これをさらに20倍増駅した放を検抜と し、包書信益機により、設定した相対過度下に おいて、権威が逸出する木分乗を、位置に至つ た鈴点で重量後によつて求めた。 鉄品としては 、ピロリドンカルポン酸ナトリウムの88合有

(個 北 桜) 的复数自抽制物含有化量本の使用感命チスト

	* *	5 60		***	AF
	***	0	2.	32	8
3 9 6	# 20	0	20	19	1
	nom.	٥	6	31	3
果のサッパリさ		۰	5	14	21.
	200	17	20	3	0
E024245	B 20	0	8	12	20

(安全性)

本希明による独出者における安全性について は、そのスタート原界が納豆であり、食用に食 されており、何ら問題はないものと推定された が、之のために、マウスによる紙口技与により 、実施例1及び4において得られた、仮白質を 主体とする抽出物について、これを特額水で分 数させて、 粘度を約3 0 epe 制近に興奮した落 解療をもとに実施したが、LD₅₀ 値は40㎡以 上であり、何ら問題なく安全性が高いものであ ると評価された。一方、一次制敵性募集につい

ては、終紀の洛解放をもとに、48時間のパツ チチストを、前送した使用感応テストと同一の 女性40名を対象に実施したが、紅葉などの異 状は弱められなかつた。

・さらに、注目されることは、実施費1中実施 例もで待られたところの、蛋白質を主体とする 拍出物には、保護潜性作用と共に、チロジナー ~ 店性の国客作用があり、さらに、この点に異 味をもつて、実施例1~4で得られた、それぞ れの抽出物において、道駄を行つたところ、実 **高男と以外で得られた益出者には、いずれもそ** の作用があることがわかつた。

したがつて執豆益品物は、保護措施作用と共 に、肌の美白的効果も関格出来るものとして有 利なものである。第3表は、本発明の実施例1 ~して得られたところの前目前沿着が示す、メ テニン石色は東生真御製作用について、インピ トロにおける政統結果を示したものである。

試験における反応系は、レーチロジン (1.0 サノビ) 0.5 ビ、リン教養者数(pH4.6) 2.0

計画電58-225893 (ア)

M、 無智水又は阻害刺液(物出物) 2.0 ml、Cmitイオン(15 ml液) 0.0 ml、 チロジナーゼ(1 ml/ml) 1.0 mlにより、 3 7.5 D の低温槽中 6 O 分の反応を進行させ、終了後に分先々度計 6 4 0 mm の耐光度を求め、生成率を禁出した。比較後体としては、アスコルビン酸(ビタオンの)を刈いた。

(前3表) 的引抽出他のメラニン生成抑制作用

単体(仮容別原:ちは、食中の食有量)					
# # #		0			
29420	Q. B	97.0			
実施男士による領白主体の他的権	5. 0	4 2. 1			
。 とによるフィトステロール主体の製品を	3 <u>.</u> 0	12.0			
。 3によるイソファボン化合物会体の助出物	0, 1	68.0			
。 4による音句質主体の連出者	2.0	47.4			
→ 1 ~ 3 で得られた抽出物の収益比率で賃金したもの	B. 0	4 5. 4			

さらに、実施例 4 で得られたところの抽出物の影像を示は、これを用幹情解させて用いると、一般と感触のよい、滑性のあるフィリングが得られることである。

アロュ多着体型束(ベラゲル―200)

••••

ピタミン 0 ・・・・・・・・・・・ 3~4 まり イノール (最粉末化品)・・・・ 0.6~8.7

一方、実施例 1 ~ 4 の抽出工程では、あらか じめ加熱又はエテレンオキサイドガスによる、 繊維処理した後の納豆を用いて、抽出処理操作 つまり、乾燥器水を、単独か又は割の粉水状の化粧料基剤と連合しておき、これとは繋に水溶液又は、医知の化粧水や乳液、あるいはより一人などをもちいて、溶解させて用いる方法である。以下に示すような、粉末化粧料を り、化粧水などを使用する際に、手の平などで乗って溶解させて用いるものである。

(粉末化粧料)

- (1) 実施例もで得た乾燥粉末・・・・ 1~10% ビタミンロ・・・・・・・・・・・・・ 0.3~30 デンプンをもつて全量100となす。
- (2) 実施例 4 で得た乾燥粉末 **** 90~955 0 M 0 又はアルギン酸 ***** 5~10
- (4) 実施例4で得られた乾燥粉末 ・・・・・ 185

に入つているも、あらかじめ被害結婚しない所 豆を用いて、抽出することも出来る。その際は 、抽出結婚工程においては、なるべく200 後又は、それ以下の高度で抽出処理を行い、 くに、持られた蛋白質を主体とする抽出もこと いては、これを被圧下で過難して粉末とする。 まではない。これを被圧でであれたのち、 を正程において、粉末となしたのち、 また、 また、 また、 はないことがわかった。

4.前型の簡単な製明

終1回は、実施例1又は4で得られた、約点から蛋白質を主体とする処出物の、水溶液としたもので、粘度が約3~6 ope 財産に調整した状態にあるものの、保養作用を示すがラフ。

1は、本副独出物の希釈旅

2は、ピロリドンカルボン使ナトリウムのS 多含有水溶液。

> 特許出職人 一鬼ファルコス株式会! (代妻者)安 斯 和

科斯司58-225803 (8)

